Serial Number: Unknown Filing Date: Herewith

Title: BIRCH BARK EXTRACT PROCESSING UTILIZING AZEOTROPIC DISTILLATION

IN THE CLAIMS

Please cancel claims 1-71 and add new claims 72-91.

- 72. A method for obtaining betulin from birch bark, the method comprising:
 - (a) contacting birch bark with an aromatic hydrocarbon solvent to provide a first mixture;
 - (b) separating the birch bark from the solvent to provide a first extract;
 - (c) contacting the first extract with an aqueous base to provide a second mixture;
- (d) heating the second mixture in solvent that: (i) is water-immiscible, (ii) is capable of forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C; effective to distill off water present in the second mixture, thereby providing a third mixture;
 - (e) separating solids from the third mixture to provide a fourth mixture;
 - (f) contacting the fourth mixture with a binder to provide a fifth mixture;
 - (g) separating solids from the fifth mixture to provide a mother liquor; and
- (h) concentrating the mother liquor, precipitating betulin from the mother liquor, crystallizing betulin from the mother liquor, or a combination thereof, to provide the betulin.
- 73. The method of claim 72 wherein the betulin is obtained in a purity of at least about 95 wt.%.
- 74. The method of claim 72 wherein the mother liquor comprises lupeol.
- 75. The method of claim 72 wherein, in step (e), the solids separated from the third mixture comprise betulinic acid.
- 76. The method of claim 72 wherein the birch bark employed comprises inner birch bark.
- 77. The method of claim 72 wherein the birch bark employed comprises outer birch bark.

PRELIMINARY AMENDMENT

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78. The method of claim 72 wherein the birch bark employed comprises Betula papyrifera,

Betula pendula, or a combination thereof.

79. The method of claim 72 wherein the aromatic hydrocarbon solvent is substituted with one

to six (C_1-C_6) alkyl, halo, or trihalomethyl groups.

80. The method of claim 72 wherein the aromatic hydrocarbon solvent is xylenes, o-xylene,

m-xylene, p-xylene, toluene, benzene, or a combination thereof.

81. The method of claim 72 wherein the solvent that: (i) is water-immiscible, (ii) is capable

of forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C, in step

(d) comprises at least one of xylene, toluene, and benzene.

82. The method of claim 72 wherein the contacting in step (a) further comprises heating the

first mixture above about 90 °C and the separating in step (b) further comprises separating the

birch bark from the solvent above about 70 °C.

83. The method of claim 72 further comprising, after step (b), concentrating the first extract.

84. The method of claim 72 wherein the fourth mixture comprises a binder selected from the

group of metal hydrides, metal alcoholates, ortho-esters and dialkoxysulfates, and combinations

thereof.

85. A method for obtaining lupeol from birch bark, the method comprising:

(a) contacting birch bark with an aromatic hydrocarbon solvent to provide a first mixture;

(b) separating the birch bark from the solvent to provide a first extract;

(c) contacting the first extract with an aqueous base to provide a second mixture;

(d) heating the second mixture in a solvent that: (i) is water-immiscible, (ii) is capable of

forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C; effective

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to distill off water present in the second mixture, thereby providing a third mixture;

- (e) separating solids from the third mixture to provide a fourth mixture;
- (f) contacting the fourth mixture with a binder to provide a fifth mixture;
- (g) separating solids from the fifth mixture to provide a mother liquor;
- (h) concentrating the mother liquor to provide crude lupeol;
- (i) washing the crude lupeol with a polar organic solvent;
- (i) recrystallizing the crude lupeol from a non-polar organic solvent; and
- (k) recrystallizing the crude lupeol from a polar organic solvent, to provide the lupeol.
- 86. The method of claim 85, wherein the polar organic solvent in (i) comprises acetone, methyl ethyl ketone, ethyl acetate, or any combination thereof.
- 87. The method of claim 85, wherein the non-polar organic solvent in (j) comprises cyclohexane, hexane, hexane, hexanes, toluene, benzene, p-xylene, m-xylene, o-xylene, trifluoromethylbenzene, or a combination thereof.
- 88. The method of claim 85, wherein the polar organic solvent in (k) comprises acetone, methyl ethyl ketone, ethyl acetate, methanol, ethanol, or a combination thereof.
- 89. A method for obtaining betulinic acid from birch bark, the method comprising:
 - (a) contacting birch bark with an aromatic hydrocarbon solvent to provide a first mixture;
 - (b) separating the birch bark from the solvent to provide a first extract;
 - (c) contacting the first extract with an aqueous base to provide a second mixture;
- (d) heating the second mixture in a solvent that: (i) is water-immiscible, (ii) is capable of forming an azeotropic mixture with water, or (iii) has a boiling point of at least 100 °C; effective to distill off water present in the second mixture, thereby providing a third mixture;
 - (e) separating solids from the third mixture;
 - (f) washing the solids with water;
 - (g) neutralizing or acidifying the solids in an aqueous acid, thereby providing a fourth

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mixture;

- (h) separating betulinic acid solids from the fourth mixture;
- (i) crystallizing the betulinic acid with a polar organic solvent; and
- (j) optionally drying the betulinic acid.
- 90. The method of claim 89, wherein the acid in (g) comprises H₂SO₄, HCl, H₃PO₄, HNO₃, HNO₂, H₃PO₃, CH₃CO₂H, CF₃CO₂H, H₃SO₃, or a combination thereof.
- 91. The method of claim 89, wherein the polar organic solvent in (i) comprises CH₃OH, EtOH, PrOH, i-PrOH, BuOH, t-BuOH, sec-BuOH, C₅H₁₁OH, acetone, ethyl acetate, methyl ethyl ketone, diethyl ketone, or a combination thereof.